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METHOD FOR PREPARING CONDUCTIVE PASTE FOR INNER ELECTRODE OF MULTI-LAYERED CERAMIC ELECTRONIC COMPONENT

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to a method for preparing a conductive paste for an inner electrode of a multi-layered ceramic electronic component, and particularly to a method for preparing a conductive paste for an inner electrode of a multi-layered ceramic electronic component that enables preparation of a conductive paste in which a conductive material is dispersed with a high dispersibility while controlling the concentration of the conductive material in a desired manner.

Description of the Related Art

Recently, the need to downsize various electronic devices makes it necessary to downsize the electronic components incorporated in the devices and improve the performance thereof. Also in multi-layered ceramic electronic components, such as multi-layered ceramic capacitors, it is strongly required to increase the number of layers and make the laminated unit thinner.

When a multi-layered ceramic electronic component as typified by a multi-layered ceramic capacitor is to be manufactured, ceramic powders, a binder such as an acrylic resin, a butyral resin or the like, a plasticizing agent such as a phthalate ester, glycol, adipate ester, phosphate ester or the like, and an organic solvent such as toluene, methyl ethyl ketone, acetone or the like are mixed and dispersed, thereby preparing a dielectric paste.

The dielectric paste is then applied onto a support sheet made of polyethylene terephthalate (PET), polypropylene (PP) or the like using an extrusion

coater, a gravure coater or the like to form a coating layer and the coating layer is heated to dryness, thereby fabricating a ceramic green sheet.

Further, an electrode paste such as of nickel is printed onto the ceramic green sheet in a predetermined pattern using a screen printer and is dried to form an electrode layer.

When the electrode layer has been formed, the ceramic green sheet on which the electrode layer is formed is peeled off from the support sheet to form a multi-layered unit including the ceramic green sheet and the electrode layer. Then, a ceramic green chip is formed by laminating a desired number of the multi-layered units to form the laminated body, pressing the laminated body and dicing the laminated body.

Finally, the binder is removed from the green chip, the green chip is baked and an external electrode is formed, thereby completing a multi-layered ceramic electronic component such as a multi-layered ceramic capacitor.

At present, the need to downsize electronic components and improve the performance thereof makes it necessary to set the thickness of the ceramic green sheet determining the spacing between layers of a multi-layered ceramic capacitor to be equal to or smaller than 3 μm or 2 μm and to laminate three hundred or more multi-layered units each including a ceramic green sheet and an electrode layer.

As a result, it is required to form an electrode layer having a thickness equal to or thinner than 2 μm, for example, and in order to satisfy this requirement, it is necessary to improve the dispersibility of a conductive material contained in a conductive paste.

Specifically, in the case where the dispersibility of a conductive material contained in a conductive paste is low, the density of the conductive material in an electrode layer obtained by printing the conductive paste on a ceramic green sheet and drying the conductive paste becomes low and the electrode layer markedly contracts when the multi-layered ceramic capacitor is

baked. Therefore, in the case where thin electrode layers are formed by printing, the electrode layers become discontinuous after baking and the overlapping area of the capacitor electrodes becomes small, whereby the effective capacitance of the capacitor becomes low.

Therefore, in order to continuously form a very thin electrode layer, it is necessary to control the concentration of the conductive material contained in the conductive paste for forming the electrode layer with a high accuracy and improve the dispersibility of the conductive material contained in the conductive paste, thereby improving the density of the conductive material contained in the electrode layer obtained by printing the conductive paste on a ceramic green sheet and drying the conductive paste.

Further, a sintering inhibitor is added to the conductive paste in order to suppress sintering of the conductive paste and in the case of a multi-layered ceramic capacitor, a dielectric constituent having the same composition as that of a dielectric substance or substantially the same dielectric substance is mixed with a conductive powder as a sintering inhibitor. In order to effectively use a sintering inhibitor, it is necessary to uniformly disperse the sintering inhibitor and the conductive powder.

A conventional conductive paste is prepared by mixing a conductive powder, a sintering inhibitor and a low boiling point solvent such as methyl ethyl ketone, acetone or the like using a ball mill, thereby dispersing the conductive powder and the sintering inhibitor in the solvent, adding a high boiling point solvent such as terpineol and an organic binder such as ethylcellulose to the thus obtained dispersed product, mixing them, thereby preparing a slurry, or mixing a conductive powder, a sintering inhibitor, a low boiling point solvent such as methyl ethyl ketone, acetone or the like and a high boiling point solvent such as terpineol using a ball mill, thereby dispersing the conductive powder and the sintering inhibitor in the solvents, adding a high boiling point solvent such as terpineol and an organic binder such as ethylcellulose to the thus obtained dispersed product, mixing them,

thereby preparing a slurry, evaporating the low boiling point solvent using an evaporator or the like to remove the low boiling point solvent from the slurry, thereby preparing a conductive paste, adding a high boiling point solvent such as terpineol to the thus obtained conductive paste in order to adjust the viscosity of the conductive paste and dispersing the conductive powder and the sintering inhibitor in the solvents using an automatic mortar or a three-roll mill.

BRIEF SUMMARY OF THE INVENTION

Problems to Be Solved by the Invention

However, in the case where a conductive paste is prepared in accordance with such a method, it is difficult to accurately control the amounts of the low boiling point solvent that have and have not been evaporated when evaporating the low boiling point solvent and it is extremely difficult to prepare a conductive paste containing a desired concentration of a conductive material. Therefore, it is extremely difficult to form an inner electrode layer having a desired dry thickness by printing the conductive paste on the ceramic green sheet. On the other hand, in the case where a conductive paste is prepared by evaporating a low boiling point solvent and the viscosity of the conductive paste is adjusted by adding a high boiling point solvent such as terpineol to the conductive paste, so-called solvent shock occurs. Specifically, the conductive powder agglutinates owing to the mixing of solvents having different affinities for the conductive powder and the sudden change in the solids concentration. As a result, it is sometimes impossible to obtain a conductive paste in which the conductive material is dispersed with a high dispersibility.

It is therefore an object of the present invention to provide a method for preparing a conductive paste for an inner electrode of a multi-layered ceramic electronic component that enables preparation of a conductive paste in which a

conductive material is dispersed with a high dispersibility while controlling the concentration of the conductive material in a desired manner.

Means for Solving the Problems

The above object of the present invention is accomplished by a method for preparing a conductive paste for an inner electrode of a multi-layered ceramic electronic component comprising a kneading step of kneading a conductive powder, a binder and a solvent to form a clay-like mixture and a slurrying step of adding the same solvent as that used at the kneading step to the mixture obtained by the kneading step to lower the viscosity of the mixture, thereby slurrying the mixture.

According to the present invention, since the concentration of the conductive material contained in the conductive paste depends upon the amount of the solvent added to the mixture, it is possible to prepare a conductive paste containing a desired concentration of a conductive material.

Further, according to the present invention, since the same solvent as that used at the kneading step is added to the mixture in order to adjust the viscosity of the conductive paste, it is possible to reliably prevent so-called solvent shock from occurring and therefore, a conductive paste containing a highly dispersed conductive material can be prepared.

In a preferred aspect of the present invention, the conductive powder, the binder and the solvent are kneaded until the mixture reaches the wetting point (ball point) thereof.

In a preferred aspect of the present invention, the conductive powder, the binder and the solvent are kneaded until the solids concentration of the mixture reaches 84 to 94%.

In a preferred aspect of the present invention, the conductive powder, the binder and the solvent are kneaded using a mixer selected from a

group consisting of a high speed shearing mixer, a planetary type kneading machine and a kneader.

In a preferred aspect of the present invention, the method for preparing a conductive paste for an inner electrode of a multi-layered ceramic electronic component further comprises a step of continuously dispersing the slurry obtained by the slurrying step using a closed type emulsifier, thereby preparing a conductive paste.

According to this preferred aspect of the present invention, since the slurry is continuously dispersed using a closed type emulsifier, thereby preparing a conductive paste, it is possible to further improve the dispersibility of the conductive material contained in the conductive paste and control the concentration of the conductive material contained in the conductive paste in a desired manner.

Further according to this preferred aspect of the present invention, since the slurry is continuously dispersed using an closed type emulsifier, thereby preparing a conductive paste, it is possible to suppress change in the solids concentration of the slurry at the dispersing step and markedly improve the efficiency of manufacture of the conductive paste in comparison with the case where the slurry is dispersed using a three-roll mill to prepare a conductive paste.

Technical Advantages of the Invention

According to the present invention, it is possible to provide a method for preparing a conductive paste for an inner electrode of a multi-layered ceramic electronic component that enables preparation of a conductive paste in which a conductive material is dispersed with a high dispersibility while controlling the concentration of the conductive material in a desired manner.

DETAILED DESCRIPTION OF THE INVENTION

In the present invention, it is preferable for the conductive powder, the binder and the solvent to be kneaded until the mixture reaches the ball point (wetting point) thereof and it is more preferable for the conductive powder, the binder and the solvent to be kneaded until the solids concentration of the mixture reaches 84 to 94%.

In the present invention, it is preferable for the conductive powder, the binder and the solvent to be kneaded using a high speed stirring device.

In the present invention, it is more preferable for the conductive powder, the binder and the solvent to be kneaded using a mixer selected from a group consisting of a high speed shearing mixer, a planetary type kneading machine and a kneader.

In the present invention, as the high speed shearing mixer, a "Henshel Mixer" (Product Name) manufactured by Mitsui Mining Co., Ltd., a "Eirich Mixer" (Product Name) manufactured by Nippon Eirich Co., Ltd. and the like are preferably employed and when the conductive powder, the binder and the solvent are kneaded using the high speed shearing mixer, the number of revolutions of the high speed shearing mixer is normally set to 500 r. p. m. to 3000 r. p. m.

In the present invention, as the planetary type kneading machine, a planetary mixer which is a planetary type mixing machine/kneading machine having two or more shafts is preferably employed and when the conductive powder, the binder and the solvent are kneaded using the planetary mixer, the planetary mixer is operated at a low speed equal to or lower than 100 r. p. m., thereby kneading the conductive powder, the binder and the solvent.

In the present invention, when the conductive powder, the binder and the solvent are kneaded using the kneader, the kneader is operated at a low speed equal to or lower than 100 r. p. m., thereby kneading the conductive powder, the binder and the solvent.

In the present invention, it is preferable to add 0.25 to 1.7 weight parts of the binder and 3.0 to 15.0 weight parts of the solvent to 100 weight parts of the conductive powder and knead the conductive powder, the binder and the solvent until the solids concentration of the mixture reaches 84 to 94% and it is more preferable to add 0.5 to 1.0 weight parts of the binder and 2.0 to 10.0 weight parts of the solvent to 100 weight parts of the conductive powder and knead the conductive powder, the binder and the solvent until the solids concentration of the mixture reaches 85 to 92%.

In the present invention, it is preferable to dissolve the binder into the solvent, thereby preparing an organic vehicle, add 3 to 15 weight % of the organic vehicle to the conductive powder and knead the conductive powder, the binder and the solvent.

In the present invention, it is preferable to add a dispersing agent to the mixture obtained by the kneading step, thereby slurrying the mixture.

In the present invention, it is more preferable to add 0.25 to 2.0 weight parts of the dispersing agent with respect to 100 weight parts of the conductive powder to the mixture obtained by the kneading step, thereby lowering the viscosity of the mixture, and then add the solvent to the mixture, thereby slurrying the mixture.

In the present invention, it is preferable to add a dispersing agent to the mixture obtained by the kneading step and slurry the mixture until the solids concentration of the mixture becomes 40 to 50% and the viscosity of the mixture becomes several pascal to several dozen pascal.

In the present invention, it is preferable to further continuously disperse the slurry obtained by the slurrying step using a closed type emulsifier, thereby preparing the conductive paste.

In the present invention, it is more preferable to further continuously disperse the slurry obtained by the slurrying step using a homogenizer or a colloid mill, thereby preparing the conductive paste.

The binder used in the present invention is not particularly limited but it is preferable to use a binder selected from a group consisting of ethylcellulose, polyvinyl butyral, acrylic resin and the mixture thereof as the binder in the present invention.

The solvent used in the present invention is not particularly limited but it is preferable to use a solvent selected from a group consisting of terpineol, dihydroterpineol, butyl carbitol, butyl carbitol acetate, terpineol acetate, dihydroterpineol acetate, kerosene and mixtures thereof as the solvent in the present invention.

The dispersing agent used in the present invention is not particularly limited and a polymer type dispersing agent, a nonionic dispersing agent, an anionic dispersing agent, a cationic dispersing agent or an ampholytic surfactant can be used in the present invention. Among these, a nonionic dispersing agent is preferable and a polyethyleneglycol system dispersing agent whose hydrophile-liophile balance (HLB) is 5 to 7 is particularly preferable in the present invention.

The conductive paste prepared in accordance with the present invention is printed using a screen printing machine or the like on the surface of a ceramic green sheet in a predetermined pattern, thereby forming an electrode layer.

Then, a dielectric paste is printed using a screen printing machine or the like on the surface of a ceramic green sheet in a complimentary pattern to that of the electrode layer printed on the surface of the ceramic green sheet, thereby forming a spacer layer and a multi-layered unit including the ceramic green sheet, the electrode layer and the spacer layer is fabricated by peeling off a support sheet from the ceramic green sheet.

It is possible to print the dielectric paste on the surface of a ceramic green sheet using a screen printing machine or the like in a complimentary pattern to that of an electrode layer, thereby forming a spacer layer, and print a conductive paste prepared in accordance with the present invention on the surface of a

ceramic green sheet using a screen printing machine or the like after drying the spacer layer, thereby forming the electrode layer.

Further, it is possible to form a ceramic green sheet on the surface of a first support sheet, print a conductive paste prepared in accordance with the present invention on the surface of a second support sheet, thereby forming an electrode layer, print the dielectric paste on the surface of the second support sheet in a complimentary pattern to that of the electrode layer, thereby forming a spacer layer, transfer an adhesive layer formed on the surface of a third support sheet onto the surface of the ceramic green sheet or the surfaces of the electrode layer and the spacer layer and bond the ceramic green sheet and the electrode layer and the spacer layer via the adhesive layer, thereby fabricating a multi-layered unit.

A desired number of the thus fabricated multi-layered units are laminated and pressed to fabricate a laminated body and the thus obtained laminated body is diced, whereby a ceramic green chip is fabricated.

Further, the binder is removed from the green chip, the green chip is baked and an external electrode is formed, thereby completing a multi-layered ceramic electronic component such as a multi-layered ceramic capacitor.

WORKING EXAMPLES

Hereinafter, a working example and a comparative example will be set out in order to further clarify the advantages of the present invention.

WORKING EXAMPLE

A conductive paste was prepared in the following manner so that the concentration of a conductive material contained in the conductive paste was 47 weight %.

1.48 weight parts of $(BaCa)SiO_3$, 1.01 weight parts of Y_2O_3, 0.72 weight part of $MgCO_3$, 0.13 weight part of MnO and 0.045 weight part of V_2O_5 were mixed, thereby preparing an additive powder.

150 weight parts of acetone, 104.3 weight parts of terpineol and 1.5 weight parts of polyethylenglycol system dispersing agent were added to 100 weight parts of the thus prepared additive powder to prepare a slurry and the additives contained in the slurry were pulverized using a pulverizer "LMZ0.6" (Product name) manufactured by Ashizawa Finetech Co., Ltd.

When the additives contained in the slurry were to be pulverized, ZrO_2 beads having a diameter of 0.1 mm were charged into a vessel so as to occupy 80 volume % of the vessel, a rotor was rotated at the circumferential velocity of 14 m/min and the slurry was circulated between the vessel and a slurry tank until holding time of the whole slurry became 5 minutes, thereby pulverizing the additives contained in the slurry.

The median diameter of the additives after pulverization was 0.1 μm.

Then, acetone was evaporated using an evaporator and removed from the slurry, thereby preparing an additive paste in which the additives were dispersed in terpineol. The concentration of the additives contained in the additive paste was 49.3 weight %.

Further, 19.14 weight parts of a $BaTiO_3$ powder manufactured by SAKAI CHEMICAL INDUSTRY CO., LTD and having a particle diameter of 0.05 μm and 1.17 weight parts of the additive paste were added to a nickel powder manufactured by Kawatetsu Industry Co., Ltd. and having a particle diameter of 0.2 μm and a mixture was kneaded using a planetary mixer for five minutes. The number of revolutions of the planetary mixer was set to 50 r. p. m.

Then, 8 weight parts of polyvinyl butyral (degree of polymerization: 2400, butyral degree: 69%, degree of acetalization: 12%) was dissolved in 92 weight parts of terpineol at 70°C, thereby preparing a 8% solution of an organic vehicle. The thus prepared organic vehicle solution was gradually added to and

mixed into a mixture of the nickel powder, the $BaTiO_3$ powder and the additive paste until the mixture of the nickel powder, the $BaTiO_3$ powder and the additive paste became clay-like and the load current value of a kneader which once became extremely high decreased and became stable at a constant value.

When the mixture was kneaded for thirty hours and 17.14 weight parts of the organic vehicle solution was then added to the mixture, the load current value became stable at a constant value.

Then, 1.19 weight parts of a polyethylenglycol system dispersing agent was added to the clay-like mixture to lower the viscosity of the clay-like mixture, thereby obtaining a cream-like mixture.

Further, an antistatic auxiliary agent, 2.25 weight parts of dioctyl phthalate as a plasticizing agent, 39.11 weight parts of the remaining organic vehicle solution and 32.2 weight parts of terpineol were added to the clay-like mixture, thereby gradually lowering the viscosity of the clay-like mixture.

Then, the thus obtained clay-like mixture was subjected to a dispersing treatment using a colloid mill three times under conditions of a colloid mill gap of 40 μm and revolution speed of 1800 r. p. m., thereby preparing a conductive paste.

The viscosity of the thus obtained conductive paste was measured using a rheometer manufactured by HAKKE Co., Ltd. under conditions of a temperature of 25°C and shearing velocity of 8 sec^{-1}.

Further, 1 gram of the thus obtained conductive paste was weighed out into a crucible and decrepitated at 600°C and the weight of the conductive paste after the decrepitation was measured, thereby measuring the concentration of the conductive material contained in the conductive paste.

The results of the measurement of the viscosity of the conductive paste and the concentration of the conductive material are shown in Table 1.

Further, whether or not any coarse particles and undissolved resin component were contained in the conductive paste was measured using a grind gauge.

The result of the measurement is shown in Table 1.

Next, the conductive paste was printed onto a polyethylene terephthalate film using a screen printing process and dried at 80°C for five minutes, thereby forming an electrode layer. Then, the surface roughness (Ra), the glossiness and the density of the thus obtained electrode layer were measured.

Here, the surface roughness (Ra) of the electrode layer was measured using the "SURFCORDER (SE-30D)" (Product Name) manufactured by Kosaka Laboratory Ltd. and the glossiness of the electrode layer was measured using a glossmeter manufactured by Nippon Denshoku Kogyo Co., Ltd.

On the other hand, a 12 mm round sample was punched out of the dried electrode layer and the density of the electrode layer was calculated from the weight of the sample measured with a precision balance and the thickness thereof measured with a micrometer.

The result of the measurement is shown in Table 1.

COMPARATIVE EXAMPLE

A conductive paste was prepared in the following manner so that the concentration of the conductive material contained in the conductive paste was 47 weight %.

An additive paste was first prepared in the manner of Working Example.

Then, a slurry having the following composition was dispersed for sixteen hours using a ball mill.

The conditions of the dispersing operation were set so that the amount of charged ZrO_2 having a diameter of 2.0 mm was 30 volume % of the ball

mill, the amount of the slurry in the ball mill was 60 volume % and the circumferential velocity of the ball mill was 45 m/min.

nickel powder (particle diameter: 0.2 μm)	100 weight parts
additive paste	1.77 weight parts
$BaTiO3$ powder (manufactured by SAKAI CHEMICAL INDUSTRY CO., LTD; particle diameter: 0.05 μm)	19.14 weight parts
polyvinyl butyral	4.5 weight parts
polyethylene glycol system dispersing agent	1.19 weight parts
dioctyl phthalate	2.25 weight parts
terpineol	83.96 weight parts
acetone	56 weight parts

Here, the degree of polymerization, the butyral degree and the degree of acetalization of the polyvinyl butyral were 2400, 69% and 12%, respectively.

After the dispersing operation, acetone was evaporated using a stirring device having an evaporator and a heating mechanism and removed from the slurry, thereby preparing a conductive paste.

The viscosity of the thus obtained conductive paste was measured using a rheometer manufactured by HAKKE Co., Ltd. under the conditions of a temperature of 25°C and a shearing velocity of 8 sec^{-1}.

Further, 1 gram of the thus obtained conductive paste was accommodated in a crucible and decrepitated at 600°C and the weight of the conductive paste after the decrepitation was measured, thereby measuring the concentration of the conductive material contained in the conductive paste.

The results of the measurement of the viscosity of the conductive paste and the concentration of the conductive material are shown in Table 1.

Further, whether or not any coarse particles and undissolved resin component were contained in the conductive paste was measured using a grind gauge.

The result of the measurement is shown in Table 1.

Next, the conductive paste was printed onto a polyethylene terephthalate film using a screen printing process and dried at 80°C for five minutes, thereby forming an electrode layer. Then, the surface roughness (Ra), the glossiness and the density of the thus obtained electrode layer were measured in the manner of Working Example.

The results of the measurements are shown in Table 1.

Table 1

	Viscosity of Paste	Concentration of Conductive materia (weight %)	Coarse Particles (μm)	Surface Roughness (μm)	Glossiness of Electrode layer (%)	Density of Electrode layer (g/cm3)
Working Example	6.3	47.2	none	0.06	83	5.6
Comparative Example	14.3	49.5	16	0.09	42	5.1

As shown in Table 1, it was found that the viscosity of the conductive paste prepared in accordance with Working Example was 6.3 Pa, while the viscosity of the conductive paste prepared in accordance with Comparative Example was 14.3 Pa, and that the conductive material was highly dispersed in the conductive paste prepared in accordance with Working Example.

Further, as shown in Table 1, it was found that although the concentration of the conductive material contained in the conductive paste prepared in accordance with Comparative Example was 49.5% and considerably different from 47% which was the target concentration of the conductive material contained in the conductive paste, the concentration of the conductive material contained in the conductive paste prepared in accordance with Working Example was 47.2% and substantially coincided with 47% which was the target concentration of the conductive material contained in the conductive paste.

Thus, it was found that according to the present invention, the concentration of a conductive material contained in a conductive paste could be controlled in a desired manner.

Further, while no coarse particles and undissolved resin component were detected in the conductive paste prepared in accordance with Working Example, coarse particles measuring 16 μm in diameter were detected in the conductive paste prepared in accordance with Comparative Example. It is reasonable to conclude that this was because the dispersibility of the conductive material was improved in the conductive paste prepared in accordance with Working Example.

Furthermore, as shown in Table 1, it was found that the electrode layer fabricated in accordance with Comparative Example had a higher surface roughness Ra and was poorer in surface smoothness than the electrode layer fabricated in accordance with Working Example. It is reasonable to assume that this was because the conductive paste prepared in accordance with Comparative Example contained coarse particles measuring 16 μm in diameter and was poorer in the dispersibility of the conductive material than the conductive paste prepared in accordance with Working Example.

Moreover, as shown in Table 1, it was found that both the gloss level and the density of the electrode layer fabricated in accordance with Working Example were higher than those of the electrode layer fabricated in accordance with Comparative Example. It is reasonable to assume that this was because the dispersibility of the conductive material in the conductive paste prepared in accordance with Working Example was higher than that in the conductive paste prepared in accordance with Comparative Example.

As described above, it was found from Working Example and Comparative Example that the conductive material was highly dispersed in the conductive paste prepared in accordance with the present invention and that according to the present invention, it was possible to prepare a conductive paste in which a conductive material was dispersed with a high dispersibility.

Further, it was found from Working Example and Comparative Example that the concentration of the conductive material contained in the

conductive paste prepared in accordance with the present invention substantially coincided with the target concentration of the conductive material and that according to the present invention, it was possible to control the concentration of a conductive material contained in a conductive paste in a desired manner.

The present invention has thus been shown and described with reference to a working example. However, it should be noted that the present invention is in no way limited to the details of the described arrangement but changes and modifications may be made without departing from the scope of the appended claims.

For example, in Working Example, although the clay-like mixture was dispersed using a colloid mill, it is not absolutely necessary to disperse the clay-like mixture using a colloid mill and the clay-like mixture may be dispersed using a homogenizer instead of a colloid mill.

Further, in Working Example, although the nickel powder, the dielectric powder and the additive paste were kneaded using a planetary mixer, it is not absolutely necessary to knead the nickel powder, the dielectric powder and the additive paste using a planetary mixer and the nickel powder, the dielectric powder and the additive paste may be kneaded using a kneader or a high speed shearing mixer such as a “Henshel Mixer” (Product Name) manufactured by Mitsui Mining Co., Ltd., an “Eirich Mixer” (Product Name) manufactured by Nippon Eirich Co., Ltd. or the like instead of a planetary mixer.

All of the above U.S. patents, U.S. patent application publications, U.S. patent applications, foreign patents, foreign patent applications and non-patent publications referred to in this specification and/or listed in the Application Data Sheet, are incorporated herein by reference, in their entirety.

From the foregoing it will be appreciated that, although specific embodiments of the invention have been described herein for purposes of illustration, various modifications may be made without deviating from the spirit

and scope of the invention. Accordingly, the invention is not limited except as by the appended claims.

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